Gas Journal

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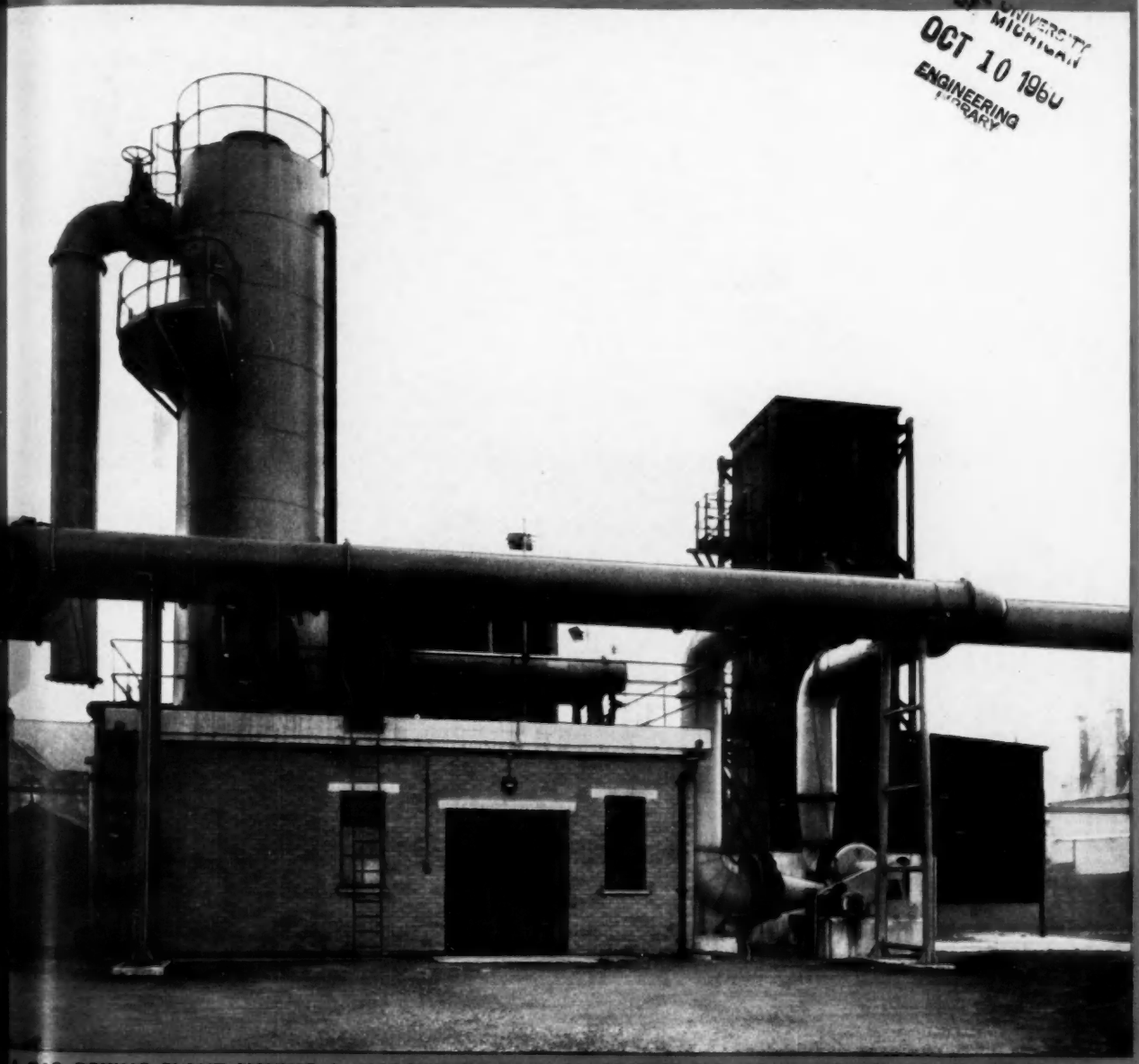

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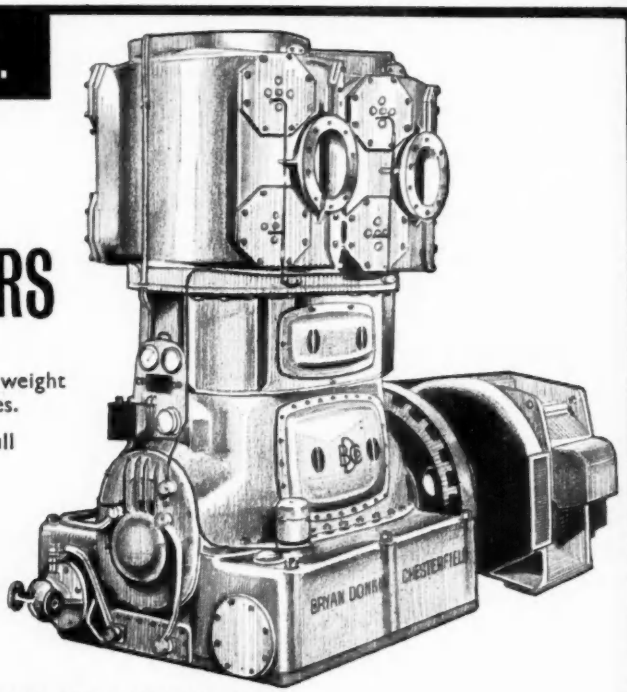

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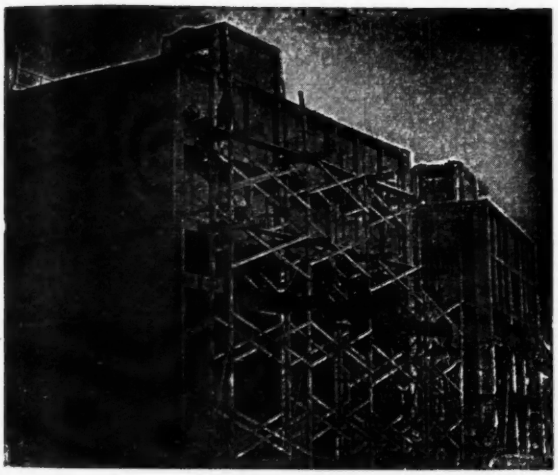

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Gas Journal

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WEDNESDAY, SEPTEMBER 14, 1960

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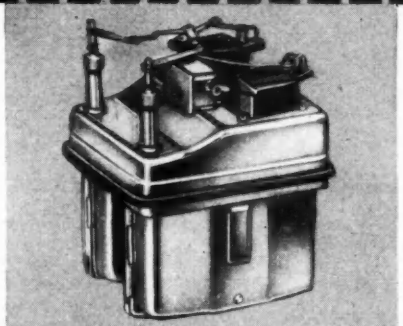

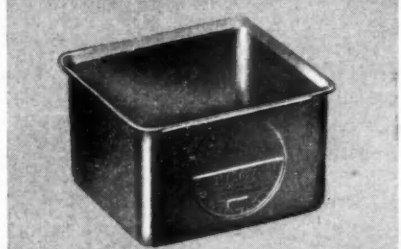

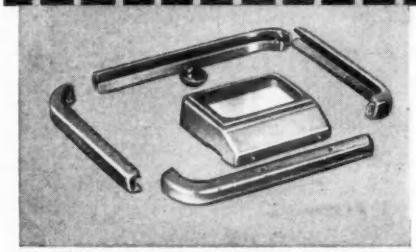

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Gas Journal

COKE OVENS • BY-PRODUCTS • CHEMICALS

The next fifty years

THE outstanding feature of the report of the study conference of 'Britain's Coal', organised last March by the National Union of Mineworkers, was the very brilliant opening paper by Mr. E. F. Schumacher, Economic Adviser to the National Coal Board, apart, that is, from the very able lecture by Dr. D. T. A. Townend under the title 'Coal and Science'.

The N.U.M., very naturally, is concerned with making a case for Government favour for the coal industry. And it is not a negligible case. Mr. S. Kessler, their Research Officer, reviewed the 'Present Situation'. Until 1956 nearly every authority was prophesying a growing demand not only for coal but for all forms of potential energy. Until then, indeed, the demand for coal, in spite of its competitors, was rising faster than supply. The mining industry was exhorted to increase its output without any limit within that of the possibilities. Oil, and later, nuclear energy, might supplement the coal supply but the foreseen growth of neither suggested a necessary curtailment of coal production.

Then came a sudden and continued fall in demand. What then, asks Mr. Kessler, had gone wrong? Mr. Kessler gave the usual six reasons. The curious thing is that at least four of these were known, or must have been foreseen, by the prophets of the earlier fifties. The economic stagnation, and later recession, and the two or three mild winters may be fairly classed as unexpected. But there was nothing unexpected about the increasing competition of fuel oil, the growth of fuel efficiency or the change of pattern of fuel consumption on the railways. And the effects of the Clean Air Act are so far negligible.

But if the prophecies have gone wrong in the short-term, there is nothing much wrong in the long-term forecasts. And in the middle-term we must accept Mr. Kessler's statement that coal will continue to supply the major part of Britain's energy needs for years to come.

There is more doubt, at any rate in some minds, about his second proposition, 'that fuel and power policy cannot be left solely to the free play of the market'. Some planning would appear to be rational but some of the manoeuvres of the N.U.M. or at least some of its members would not appear to lie in that direction.

Turning now to the long-term view the tone was set in the first paper of the conference presented by Mr. Schumacher, concluding with the impressive words 'The conviction that Nature's deposits of concentrated carbon are a unique and irreplaceable bounty, a once-for-all endowment of mankind to ease its task of living, and that no generation has the right to abandon and ruin any of them for the sake of a small, fleeting convenience'.

The fact is, of course, that in our discussions we are apt to overlook the elementary facts. We too often talk as if electricity and gas, even oil-gas, were something more than mere forms of coal (or oil) preparation for the market. And we talk, too, as if uranium or its possible substitutes were infinite in available quantity whereas, at least in accessibility, these materials are not likely to be vastly greater in availability than coal or oil.

The exasperating thing about this impressive paper, and others more or less like it which have appeared over the last, say, 30 years, is that there is little new in it. Technologists have warned the public over many years, though not in words so felicitous as Mr. Schumacher's, that 'The world's reserves of concentrated carbon deposits are of finite quantity which is not a very large multiple of the quantity that is being consumed every year'. And that from this angle, therefore, the fact of 'non-renewability' possesses the greatest possible significance.

These solemn warnings are not new. The public has just gone on blazing away at coal, at least, as if there would never be an end to it and with ridiculous efficiencies, the railways being perhaps the worst offenders of all.

Here one pauses to reflect whether the users of oil are not doing something like the same thing. Are not the luxuries of travel at supersonic velocities being purchased at a prodigious cost in oil? Shall we, at some not very distant date, have to crawl about a planet whose mineral wealth we are now squandering at a fantastic rate, and, what is worse, a rapidly increasing rate? The outsider might ask: Is there any increase in overall efficiency in gasifying a small fraction of the available oil, when it could be used for heating purposes with a little less convenience in its original liquid form?

There are many striking passages in Mr. Schumacher's address to which we can only briefly refer. Perhaps of the most immediate importance is his discussion of the concepts of 'economic' and 'uneconomic', which he shows cannot be applied to the extraction of non-renewable resources without very great caution. The closing of an uneconomic pit may simply mean the loss of valuable assets which have to be replaced when scarcity value makes it imperative to open up again. But this part of the address cannot be summarised without destroying its cogency.

Mr. Schumacher sums up what seems to him 'a reasonable and prudent view of the future' in five propositions which we suggest are irrefutable and which may be put briefly as follows:—

1. There is as yet no substitute for natural carbon as a cheap source of heat and energy.
2. Most of the world's concentrated carbon deposits are coal, though by far the largest part of the *increase* in world fuel requirements is being met by oil.
3. There is as yet no effective substitute for oil in the internal combustion engine.
4. Almost certainly before the end of the century the availability of coal may be an essential condition for the continuance of modern civilisation at least in some countries.
5. These propositions are likely to apply particularly to this country which possesses no indigenous sources of oil and natural gas.

His elaboration of these propositions is well worth studying in full.

The discussion of this remarkable address, under the able Chairmanship of Sir Geoffrey Crowther, is also not to be missed. We hope to return to it later.

Therm and variations

WHATEVER will they think of next? We have seen recipes produced in some unusual ways but never on the turntable of a gramophone. The Wales Gas Board has adopted a new idea for publishing Welsh recipes—in the form of a 45 r.p.m. plastic gramophone record called '3 Variations on a Welsh Therm.'

It was played at a Press Conference by Mr. T. Mervyn Jones, Chairman of the Wales Gas Board, who explained that the recipes had been chosen from the Board's Welsh cookery book which has gone all over the world. The record is made of plastic and is enclosed in a bright greetings folder. Mr. David Pugh, formerly of Newport, has 'translated' three recipes in verse, and they are delightfully sung by Miss Esme Lewis, to 'three phoney folk songs' specially composed for the occasion by Dr. Daniel Jones. Sleeve design is nice, too, and includes a message from 'Jones the Gas.'

Australian convention

WE do not hear a great deal about the Australian gas industry these days, although New Zealand was in the news a few years ago when the gas industry there was strongly criticised, and as strongly defended. Unhappily our news of gas in the Pacific is less regular than we would like it to be. But we do know that in both countries the industry is developing in many interesting ways. A reflection of that development is the Australian Gas Industry Convention which is to be held in Sydney on October 17-20—a convention which promises a wide range of interests.

The objectives—and here we quote the official programme—are 'to encourage the exchange of ideas which will promote and develop the gas industry in all its branches so that it may serve to the fullest the best interests of the industry and the public; to advance knowledge and stimulate further advancement in gas manufacture, marketing and service; to emphasise the important rôle modern marketing methods must play in the merchandising of gas appliances and services; to promote closer relations and co-operation between all branches of the gas industry, both in Australia and overseas, including plant and equipment makers, gas producers and marketers, appliance makers and those concerned with gas by-products.' Which seems to cover just about everything.

The convention sponsors are the National Gas Association of Australia, the New South Wales Institute of Gas Engineers and the Australian Gas Institute. The tentative programme includes a marketing forum in which three of Australia's top business brains will discuss the principles of modern marketing and their applications in the gas industry, an examination of sales training, an address on modern appliance development, papers on recent oxide purification practices and on the economic aspects of removing organic sulphur, and 'a precedent-breaking paper' on oracle production scheduling in a complex gas-making process; there will also be an address by a prominent member of the oil industry, by the presidents of the three convention sponsors, and so on. The 'outside' activities listed in the programme includes an item intriguingly labelled 'Night Life of Sydney,' the effect of which was somewhat spoiled for us by the announcement that this would include a theatre party to see 'The Merry Widow'!

An important visiting speaker will be Mr. Chester S. Stackpole (of I.G.U. and Sc.G.B. fame), Managing Director of the American Gas Association, and representation is expected from Japan, Hong Kong, the Philippines and Europe. We understand that the Institution of Gas Engineers will be represented by Mr. J. West, deputising for the President, Mr. H. Cheetham.

Spent oxide in sulphuric acid manufacture

By K. B. EVANS,
A.R.I.C.
Chief Chemist ; A. C. C. Brotherton (Birmingham) Ltd.

THERE are two main processes for producing sulphuric acid—the nitration or chamber process and the contact process. The nitration process with its raw materials of spent oxide, pyrites and zinc concentrates, depends on the use of oxides of nitrogen and complicated chemical reactions to effect the oxidation of sulphur dioxide to sulphur trioxide and its absorption in water to produce an acid. The contact process uses mainly elemental sulphur or anhydrite as raw materials and by direct catalytic oxidation of the sulphur dioxide and absorption in sulphuric acid, produces a much purer and more concentrated final product.

The lead chamber method of producing sulphuric acid commercially has been in use for over 200 years. The original raw material used was brimstone to be followed later by pyrites—a naturally occurring iron sulphide. About 110 years ago iron oxide was first used to replace lime for the purification of coal gas, and it is reported that in 1861, only 12 years later, over 2,000 tons of spent oxide was used for making sulphuric acid.

Dependence on imported sulphur

The contact process, which has been in operation now for 60 years, is largely dependent on imported sulphur as the raw material and has mainly met the increased demands for sulphuric acid in that period. Since the early 1930's increasing amounts of acid have been produced from anhydrite—an anhydrous calcium sulphate occurring naturally in this country.

At the present time about 41% of the total sulphuric acid produced in this country is derived from imported sulphur, 17% from pyrites, 17% from anhydrite and 12% from spent oxide. This 12% represents over 200,000 tons of spent oxide being burnt annually and producing about 300,000 tons of sulphuric acid (as 100%).

Following the successive sulphiding and revivification reactions that take place in the purifiers at the gasworks, the spent oxide received at the acid works contains about 49% of sulphur, present as elemental sulphur. The other major constituents and their approximate percentages in the spent oxide as received at the author's works are, water 8%, iron oxide 22% and wood 10%. The iron should all be present as the oxide since any remaining iron sulphide present, due to incomplete final revivification, could cause the oxide to catch fire during transport, crushing or storage. In addition, spent oxide normally contains Prussian blue formed from the cyanides present in the gas, tarry matter, calcium salts from the lime originally added to the fresh oxide, ammonium salts, silica and aluminium oxide. The amounts of these latter compounds present will vary with both the source of the original iron oxide used for purification and the type of gas purified. Most of them do not normally greatly affect the process of manufacturing acid from spent oxide as long as they are only present in small amounts.

One of the main advantages of most spent oxides is the absence of arsenic which enables an arsenic-free acid to be produced in contrast to the product obtained from Spanish pyrites.

The following account of the handling and combustion of spent oxide will relate to the works of a medium sized chemical manufacturer working in close association with an area gas board.

The spent oxide is mainly received in rail wagons from which it is unloaded by grab directly on to a belt feeding it into a single roll crusher to break up any large lumps present. Next the oxide passes through a swing hammer crusher to reduce the size further and is then carried up a bucket elevator and discharged into a storage hopper. Mention may be made here of the sampling of the oxide, as it is at the top of the bucket elevator where this is carried out. As the oxide is tipped from the buckets there is a small sampling spoon rotating at about 12 r.p.m. in the oxide stream. This collects a small sample of oxide from about every eighth bucket and throws it into a chute from which it falls down a pipe into a bin at floor level. The binful can then be further reduced to give a suitable sample for laboratory testing.

From the storage hopper the oxide is carried by a conveyor belt to a second hopper in the burner house. At one point above this conveyor an electromagnet is suspended to remove any pieces of metal which could cause damage to the burners. When required spent oxide is discharged from this hopper into skips, weighed, and then tipped into the small burner feed hoppers. These are fitted with a grid on top to remove any large lumps which may have escaped the crushers. The feed hoppers have a rotating feed plate at the base which gives a controlled feed rate into a small bucket elevator which tips the oxide into the top of the burner.

Burning spent oxide

The furnaces in which the spent oxide is burnt are Harris burners. These are constructed of brick, braced externally by steel work and are circular in cross section. They consist of three hearths one above the other and the spent oxide falls on to the top hearth near the centre. The burner has a central rotating shaft which carries a single arm on each hearth. The arms are fitted with rakes set at angles so that the burning spent oxide is evenly distributed over the surface and also slowly pushed across the hearth. Thus the oxide is moved from the centre to the outside of the top hearth where it falls on to the second hearth. Here the rotating arm and rakes move it around and across to the centre of the burner where it falls to the third hearth. After travelling across the third hearth the burnt residue leaves the burner.

Spent oxide when ignited burns readily, and once a burner has been started no further external heat is required to maintain combustion. Air for combustion is drawn into the burner and the gases are taken off by a fan placed further on in the chamber plant system. Thus the burners operate under a very slight suction which

CHEMICALS AND BY-PRODUCTS

eliminates any gas passing out into the burner house. Gas uptakes are fitted between the hearths in such positions as the air drawn in at the base has to pass across each hearth in turn and up to the one above. Temperatures of over 900° are reached on the top hearth, and the arms and rakes are made of a chrome alloy steel to combat the effect of the reducing sulphurous atmosphere at this temperature. In addition, the centre shaft is hollow and is air-cooled from a fan. This cooling air also circulates through the rotating arms.

In the combustion of spent oxide the sulphur present burns to sulphur dioxide together with the formation of some sulphur trioxide. On the top hearth some of the sulphur will volatilise and burn in the air present, but it is probable that at this temperature and with the excess of sulphur present the iron oxide will re-combine with some to form iron sulphide which will burn to iron oxide and sulphur dioxide later in the furnace. This is borne out by the fact that often the top hearth of the burner gets built up with a fused material which analysis shows to be mainly iron sulphide together with some silica and calcium salts. Occasionally when this fused material has built up to too great a depth the burner has to be shut down to enable it to be cut up off the hearth.

Considering the effects of the substances present other than sulphur and iron oxide, the wood will burn to carbon dioxide and water vapour, and its presence tends to raise the temperature of burning. The moisture will simply vaporise in the burner, and if the oxide is too wet this throws an extra load on the Glover tower, one of the main purposes of which is to drive off water from weaker acids produced in the chambers, and concentrate them. The Prussian blue will decompose and produce carbon dioxide, nitrogen and iron oxide. The tars and oils will vaporise and burn, but if present in too great a quantity will not be completely burnt and may pass forward and affect the quality of the product. The calcium present has a disadvantage in that it will fix some sulphur as sulphate which will remain in the burnt residue, thus reducing the efficiency of sulphur recovery. Ammonium salts must not be present to a great extent, since although small amounts would decompose, larger amounts would mean ammonia gas passing forward and adversely affecting the acid-making process. The aluminium oxide would be unaffected by the burning but, although even if present in very small amounts, a large proportion of it tends to be carried forward in the gases and appears in the final product. Here it can be troublesome in some processes for which the acid is required.

Another type of furnace used for spent oxide is the Flixborough rotary burner. This consists of a revolving cylindrical brick-lined shell inclined very slightly to the horizontal and containing a central steel tube. The oxide is fed in at one end of the outer shell together with the air for combustion. The burnt residue is discharged from the lower end, but the hot gases are drawn along the central tube back to the feed end of the burner, where they preheat the incoming oxide and air. This burner gives a lower temperature of burning and also ensures that the tar and oils present in the spent oxide are completely burnt in the central tube which acts as a secondary combustion chamber.

The gases passing forward from the burners contain 6 to 7% sulphur dioxide and 10% of oxygen from the excess air drawn in through the burners, which is necessary for the acid-making reactions following. The carbon dioxide, approximately 3%, and the nitrogen from the air used, are simply diluents and take no part in the reactions in the plant.

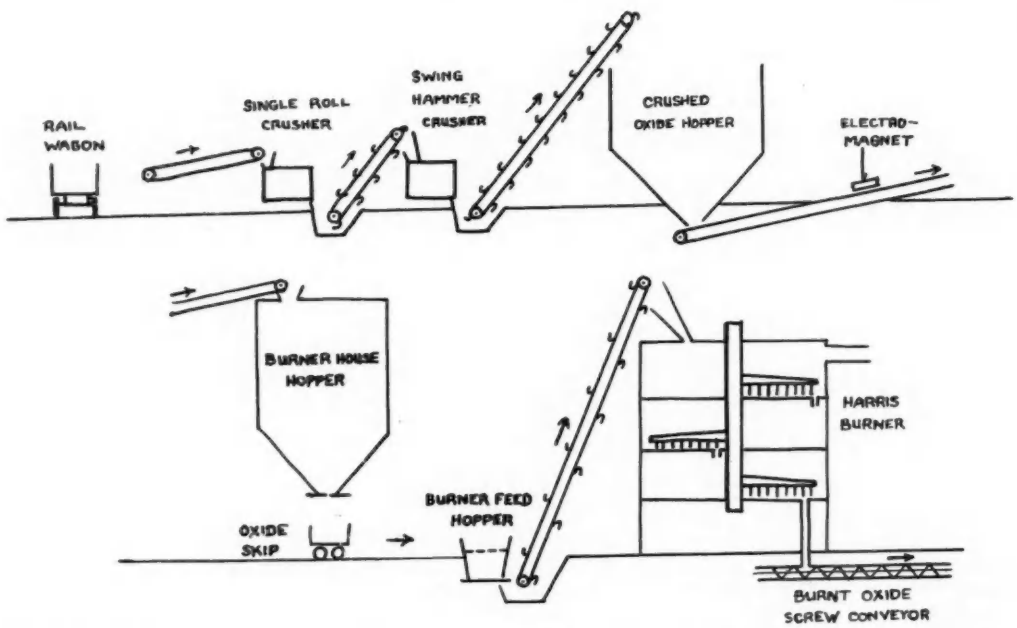

Diagram showing the mechanical handling arrangements for spent oxide from railwagon to the burner.

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The recovery of pure sulphur from coal gas and fuel gases

By J. J. PRIESTLEY,
M.Sc., M.Inst.Gas E., A.M.I.Chem.E.
Research and Development Department, W. C. Holmes & Co. Ltd.

THE element sulphur resembles in a way the ancient Roman deity Janus, who had two faces looking in opposite directions. It is an essential commodity to civilised life as we know it, yet at the same time it is the major source of corrosion and ill health in our cities due to atmospheric pollution by fuel combustion. Town gas is the only combustible fuel from which virtually all sulphur is removed prior to burning. Although the consumption of electricity does not generate sulphur bearing gases, there is considerable production during generation.

The removal of sulphur from town gas is a legal liability as far as hydrogen sulphide is concerned (and the date may not be far distant when a restriction is placed upon the organic sulphur content). An ideal state of affairs would include not only the removal of sulphur from town gas, thereby eliminating a nuisance, but also selling the recovered sulphur at a profit, thereby creating a benefit. Unfortunately sulphur recovered in this way has to compete against imported sulphur in one form or another, and whether or not sulphur removal is profitable depends on world prices of sulphur.

Another uncomfortable factor which puts the traditional iron oxide method of sulphur removal at a disadvantage is the change in the method of making sulphuric acid. Formerly most of the sulphuric acid production was by the lead chamber process which utilised pyrites and spent oxide. Modern catalytic processes are designed to use pure sulphur or 'brimstone.' One of the major problems facing the gas industry arising out of this change is hardly one of making a profit or even a loss but one of the physical disposal of large amounts of spent oxide. The production of sulphur from the Lacq natural gas field has accelerated the deterioration of the situation.

The possible solutions to this impasse, apart from dumping spent oxide, would appear to be either the extraction of pure sulphur from spent oxide, or alternatively the removal of hydrogen sulphide from the crude gas by alternative methods which would yield pure sulphur.

Pure sulphur from spent oxide

The extraction of pure sulphur from spent oxide is a well established process both in this country and more extensively in Germany. In German practice, oxide purifying material is fouled up until it contains about 33% by weight (D.B.) of sulphur, rarely more, after which it is first given a simple water wash, the object of which is to reduce the concentration of soluble salts. The washed oxide is then dried in a rotary drier, to a moisture content of 10%. It is then loaded into flat circular baskets, each of which holds one ton of material, the base of the basket being made of perforated metal. A number of these, when charged with oxide, are superimposed inside a steel cylindrical extractor, which is sealed and then slowly flooded with carbon disulphide. The flow of carbon disulphide actually passes through a number of extractors in series, arranged so that the fresh solvent enters the unit which has been extracted for the longest time, and leaves from the most recently charged unit, thereby achieving a counter-current effect.

A fully extracted unit is taken off stream, drained and steamed out to drive off the final traces of carbon disulphide, after which the extracted oxide is removed and used as the basis of fresh purifying material. Sawdust and calcium carbonate or sodium carbonate are added and the whole given a thorough mixing.

The solution of crude sulphur in carbon disulphide is next given two successive washes with concentrated sulphuric acid, then a soda wash and finally a water wash. This treatment removes the organic material, such as tar and polymerised gums, which would foul and discolour the sulphur. The washed solution is then sent to a batch still in which the carbon disulphide is evaporated. Towards the end of the distillation the temperature rises above the melting point of sulphur, enabling it to be run out as a clear light-brown liquid into concrete bays where it cools and crystallises.

Risk of fire

Stringent precautions are taken against fire risk, the extraction plant being built on pillars in a sunken compound flooded with water. All vents are taken to a central scrubber and washed with oil, from which the solvent is recovered by distillation. The sulphur produced is bright yellow and is at least 99.95% pure. The recovered oxide may be used three to four times before it is finally rejected. On a large British installation of this type a soda wash is used instead of the preliminary water wash, with the object of removing 'blues' or iron cyanides. This improves the economics of the process as a whole, the recovery of sulphur alone being uneconomic.

There is no process in use which produces pure sulphur direct from crude coal gas. Those using aqueous solutions as a purification medium instead of aqueous suspensions produce a better product, but even this rarely exceeds a 97% or 98% purity, which is not good enough. Even this quality of sulphur is black in appearance, as very little impurity is needed to cause a marked discoloration. Liquid purification solutions dissolve traces of tar and gummy bodies from the gas which are possibly adsorbed by the precipitated sulphur and are responsible for the poor colour and quality of the recovered product.

As an example, the Thylox process uses thioarsenate solutions, the washing liquid being cycled between a series of washing towers or absorbers on the one hand and oxidisers on the other. Sulphur appears as a froth on the surface of the oxidisers from which it is taken to rotary filters, where it is separated from any entangled solution, and further is water washed to remove as much of the arsenical solution as possible. The recovery of a pure

Continued on p. 408

Sulphur extraction from spent oxide by solvents

By G. J. MIDDLETON,
Assoc.M.Inst.Gas E.
Sales Engineer, Chemical Plant, Newton, Chambers & Co. Ltd.

THE introduction of the Gastechnik process and the concentration of gas manufacture have tended to renew interest in the recovery of sulphur in the elemental state from spent oxide. Recovery of solid sulphur from spent oxide has been practised in England and on the Continent for several decades, but the relative quantity of oxide so treated today in England is very small.

The economics of gas purification with spent oxide are considerably influenced by oxide costs, not the least of which is the disposal of spent oxide. In many areas today, an appropriate value for the sulphur content of spent oxide cannot be obtained, whereas the sulphur, if extracted, may be sold in accordance with the prevailing market conditions.

It is the purpose of this article to review the available solvent processes and to indicate their potential application to the gas industry as now constituted.

The sulphur obtained from the spent oxide must preferably compete with imported pure sulphur so that the current market price may be obtained. Hence (1) it must not be less than 99.8% pure, and (2) it must be yellow and clean looking. Any discoloration of the sulphur would appreciably reduce its acceptance by the market.

Any extraction system should enable as great a proportion of the oxide as possible to be returned to the gas works for further use in purification. This requires that the oxide be reconditioned and ideally this would comprise:

1. Means to remove the permanent salts resulting from side reactions during the course of gas purification, *e.g.*, Prussian blue, thiocyanates, thiosulphates, sulphates, etc.
2. Adjustment of the water content.
3. Adjustment of the porosity.
4. Adjustment of the *p*H.
5. Adjustment of activity.

Removing permanent salts

In practice, it is difficult to achieve complete removal of the permanent salts, but on at least one large installation in Germany, a considerable reduction in the salt content is achieved by water washing the spent oxide before solvent extraction. While this procedure is relatively simple, it creates an effluent which may, or may not, be readily discharged, and requires the drying of the oxide before extraction. Ground space and handling requirements are considerable.

It is concluded therefore, that it would only be warranted in special cases, and that with the majority of oxides in use in England it would be economically advantageous to control salt content by withdrawing a portion of the oxide during each purification/extraction cycle. The withdrawn oxide would be replaced with new active material. Investigations indicate that some 10% to 15% of the oxide would require disposal per cycle, thus giving an oxide life under equilibrium conditions of some seven to ten years. At the same time, the introduction of new oxide or other form of activating agent would enable the desired level of oxide activity to be readily maintained.

The extent of the plant required and its operating cost is obviously dependent on the solvent power of the extraction liquid. At the same time the allotropic form or combination of allotropes in the sulphur present in the oxide is significant, since solubility in any chosen liquid is a function of the physical form of the sulphur.

The amorphous form of sulphur is insoluble, but it is only present in the normal run of works oxide in insignificant proportions. In the exceptional case, however, the potential difficulty due to the presence of amorphous sulphur may be readily overcome by raising the oxide temperature to 90°C., *e.g.*, with steam for a short period during which the amorphous sulphur reverts to the soluble form.

The bulk of ordinary sulphur consists of the alpha allotrope in rhombic and monoclinic forms, the latter being more soluble than the former. However, the rhombic form predominates and therefore determines the solubility characteristics.

Sulphur solvents

Typical solvents for sulphur in industry are the chlorinated compounds, trichlorethylene, perchlorethylene and chlorobenzene; the aromatics, benzene, toluene and xylene, and carbon disulphide.

Benzene at 90°C. will only dissolve 17 g. of sulphur per 100 cu.cm., whereas carbon disulphide, by far the best solvent, will dissolve 120 g. per 100 cu.cm. at 46°C. The chlorinated compounds have solvent powers in between.

The vices of the chlorinated compounds in use are now well known and need not be stressed. It is sufficient to say that their use in Germany has been discontinued for some time and there is no indication that the potential corrosion difficulties can be readily overcome.

In considering the aromatic solvents such as benzene in relation to carbon disulphide, there are toxic and inflammable properties in both cases, but carbon disulphide possesses a very much greater solvent power. Both liquids have been in large scale industrial use for generations and plant engineering requirements are equally well understood. Carbon disulphide has a greater degree of inflammability but burns with a low temperature flame and is very much denser than water so that effective fire protection measures are very easily arranged.

It will be seen, therefore, that there is no over-riding reason why the economic advantages associated with the high solvent power of carbon disulphide cannot be realised. Existing large scale plants in Germany and England take full advantage of it and have been in successful operation for over 30 years. The service requirements are approximately half those required for benzene. In the following discussion therefore it is assumed that carbon disulphide

is the chosen solvent except where specifically stated otherwise.

The sulphur in spent oxide is dispersed throughout the material and can only be removed by the penetration of the solvent followed by diffusion of the solution formed to the exterior of the mass and subsequent dispersion in the bulk of the solvent. The process is therefore in the nature of a leaching operation—diffusion is the controlling factor.

The rate of diffusion is primarily a function of time and concentration gradient. The latter cannot be large if the bulk of the solution is highly concentrated, yet for economic recovery of the sulphur from the solution, the concentration should be high. This difficulty is overcome by countercurrent multi-stage operation; for instance, in one very economical plant five stages are employed. However, even where the concentration gradient is most favourable, the extraction period amounts to several hours. A period equivalent to one shift is quite common.

After the sulphur has been leached from the oxide, it is necessary to remove the solvent still saturating the oxide. This is done by steam distillation, low pressure live steam being employed in direct contact with the oxide.

Here again diffusion is a dominant factor since the solvent has to travel from within each particle of the oxide and from within each particle of the remaining sulphur. Solvent volatility is also important—particularly in relation to the grade of steam that may be employed. Once again countercurrent multi-stage operation is necessary for maximum economy, and in the above-mentioned plant employing five extraction stages, there are six stages for steaming out the solvent.

It must not be overlooked that while a little sulphur may be retained in the extracted oxide without detriment, it is essential that the solvent be removed completely. Removing the last traces is necessarily a slow process and is quite likely to occupy two shifts.

Where carbon disulphide is employed as the solvent at near ambient temperature, a typical operating cycle is shown in Table 1.

TABLE 1.

Phase	Time taken in hours	
	loose oxide	Pellets
Extraction	8	14
Steaming	16	7
Changing oxide, etc.	1	1
Total	25	22

The sulphur content of the extracted oxide in either case is comparable, namely between 1 and 2%.

When benzene is employed in a plant of similar size and utilising the same number of stages for the extraction of oxide pellets, hot working at 90°C. is advantageous, but even so the extraction cycle may take as long as four shifts. For a given throughput, therefore, such a plant would be markedly larger than one employing carbon disulphide, even allowing for twice the steam consumption when using benzene. Aromatic solvents are not recommended for use with loose oxide which tends to disintegrate and compact into a dense mass during extraction with these liquids.

The chlorinated solvents have been applied to the extraction of oxide pellets on a different principle involving static leaching with thermal circulation. Their operation is therefore not comparable with the high efficiency systems considered above, but for details of the application of perchlorethylene to the extraction of oxide pellets in

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England, reference may be made to the paper by J. Powdrill to the Institution of Gas Engineers in 1959 entitled 'Operation of liquid and Gastechnik purification plants at Cardiff.'

The ordinary run of works oxide is sufficiently contaminated with tar and other soluble organic constituents to prevent the direct production of pure sulphur from the extract solution. Some form of purification is necessary before final evaporation of the solvent from the sulphur.

The conventional purification process—with carbon disulphide as the solvent—consists of sulphuric acid washing to break down the organic impurities, rigorous filtration to remove the carbonaceous and entrained oxide particles and finally alkali washing to remove any residual acidity. According to plant scale, batch or continuous working may be employed and the acid washing is advantageously carried out in countercurrent multi-stage equipment on the larger installations.

The removal of fine particles suspended in the solution is most important, and is best carried out by Fuller's earth. Apart from filtration, Fuller's earth exerts some decolorising effect which contributes to the appearance of the refined sulphur.

Semi-continuous distillation

To obtain the sulphur from the purified solution it is usual to distil off carbon disulphide semi-continuously by indirect steam. During a run sulphur accumulates in increasing concentration in the still until the feed is interrupted. Steam heating is continued in order to evaporate the concentrated solution to molten sulphur, all traces of carbon disulphide being finally expelled with the aid of a little direct steam. The molten sulphur is then discharged and a new run commenced.

The sulphur product may be sold in several forms, *viz.*, molten, granulated, cast, or broken. Obviously, advantage should be taken of any opportunity to sell molten sulphur since this minimises labour requirements and eliminates mechanical handling. There is a restricted market for granulated sulphur, but its production is not recommended where water contamination cannot be tolerated, since a little water is invariably associated with it even after prolonged draining and additional plant is required for its removal.

On the small scale the sulphur may be cast into easily handled bricks, but where production is greater than 2,000 tons per year or so, it is simpler to cast it in large troughs, after which it is broken up mechanically and handled as needed.

The effect of plant size is very considerable, since the larger the plant the greater the technical efficiency and the lower the overhead costs. With large plant, it is economically more feasible to achieve a high degree of countercurrent operation throughout the actual extraction process, thus markedly reducing steam, water and acid requirements. With large plant, too, it is practical to mechanise fully the whole of the oxide handling and thus minimise labour costs.

From recent cost investigations it is apparent that the production of sulphur at a commercially competitive price from loose spent oxide is unlikely to result with plants of less than 10,000 tons of sulphur per year capacity. This and greater sizes of plant would enable full mechanisation to be economically employed and the labour and operating charges would be at a minimum.

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It is obvious that this scale of operation only applies to the larger works or to a centralised installation treating the oxide from several works. The final production cost of the sulphur is, however, adversely affected by the oxide transportation charges, so a point is reached when these charges begin to outweigh the savings from increased plant capacity of the centralised extraction plant. It is, therefore, essential to assess accurately all transportation charges if its economic optimum size is to be determined.

As an example of the potentialities of this form of plant when applied to the gas industry of today, the economics of a plant designed to produce 20,000 tons of sulphur per year from loose spent oxide are considered below.

The complete installation as visualised is illustrated diagrammatically in Fig. 1. The oxide is assumed to contain 50% of sulphur and 1% of tar by weight, and the extracted oxide is reconditioned for further use, 15% of the extracted oxide being made redundant and replaced by new.

The operating requirements of such a plant are shown in Table 2.

Calculated on the basis of current prices—steam at £1 per ton, depreciation at 8.36%, maintenance at 2½%, etc. —and including depreciation of the entire installation, but excluding all oxide transportation charges, the f.o.r. price of the sulphur obtained works out at £7 12s. per ton. It will be noted that there is a reasonable difference between this production cost and the current price of pure sulphur (99.95%) to accommodate considerable oxide transportation charges.

The application of modern chemical engineering techniques to the extraction of sulphur from spent oxide by solvents has done much to reduce the overall cost of the processes, particularly if advantage is taken of the solvent power of carbon disulphide when this liquid is used as the solvent.

TABLE 2.

	Per ton of sulphur
Steam	1·9 tons
Water	10,000 gal.
Electricity	25 kw.h.
Carbon disulphide loss	·015 tons
Acid	·05 tons
Alkali	·01 tons
Labour-shift men	3
daymen	1

For maximum economy an installation should be of reasonably large capacity and it may be that the oxide from several works in one area can be economically handled by a centralised extraction plant. Where oxide disposal and oxide costs have become an excessive burden on the conventional gas purification process, the availability of plant to recondition the spent oxide with sulphur recovery should not be overlooked.

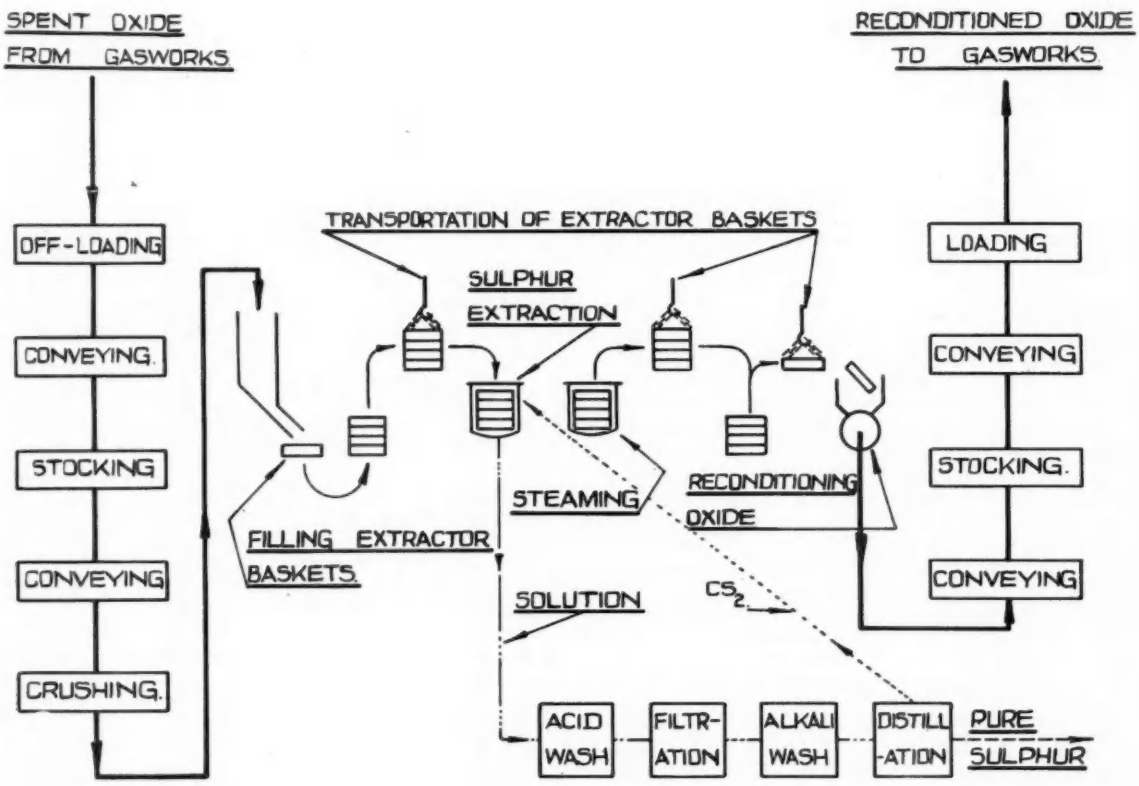

FIG. 1.

The production of concentrated ammonia and the dephenolation of effluent liquor

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THE production of concentrated ammonia liquor stems from the arrival of synthetic ammonia which has continued, in general terms, to make the production of ammonium sulphate more attractive if carried out in larger plants to which the ammonia is transported more cheaply as concentrated liquor. At the same time, the ever increasing stringency of effluent disposal requirements now demands that gasworks should condition their crude liquor to a greater or lesser extent before disposal locally.

The production of concentrated ammonia is an economic aid to the production of a satisfactory effluent when crude liquor cannot be discharged directly and it is necessary to reduce the toxity by a simple steam distillation. While subjecting the crude liquor to steam distillation, the oxygen absorption value is reduced in proportion to the hydrogen sulphide, thiocyanate and phenolic constituents removed, together with the dilution resulting from the condensation of stripping steam.

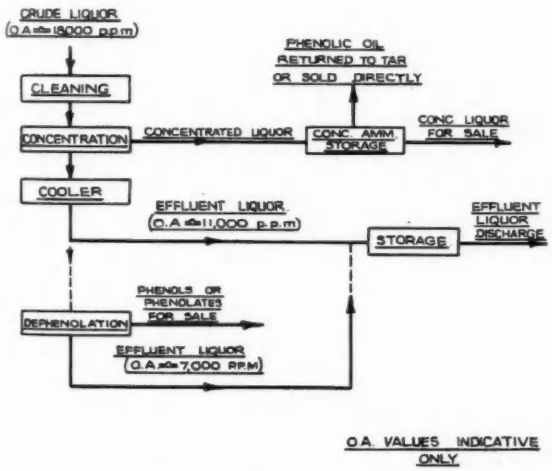

FIG. 1.

The economics of the distillation process obviously benefit if a remunerative ammonia concentrate can be obtained from the liberated vapours. It is the realisation of this which gives rise to the modern conception of the 'concentrated ammonia plant', which is considered in more detail later.

Although about one-third of the ammonia in the crude liquor is present in the 'fixed' state, its recovery in this form is wholly uneconomic because of the increased plant complexity and much increased labour costs. Fortunately, it is rare that effluent disposal requirements necessitate this step and in point of fact the toxity of the effluent is not markedly improved in terms of oxygen absorption value by the recovery of fixed ammonia.

When further conditioning of the crude liquor prior to its disposal cannot be avoided, consideration should be given to the removal of monohydric phenols and, as a further step, removal of higher tar acids. The resulting reduction in oxygen absorption value is considerable, typically 3,000 to 5,000 p.p.m. on account of monohydric phenol removal alone.

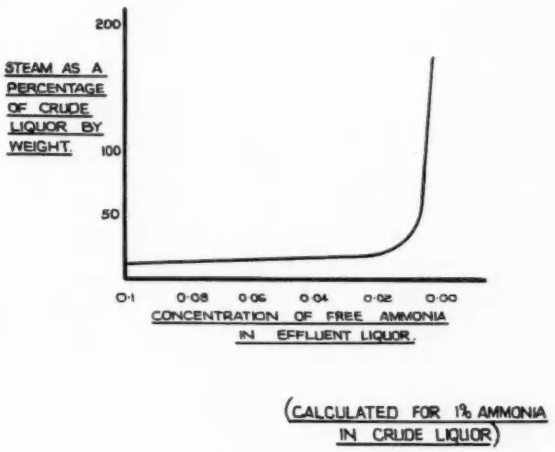

FIG. 2.

Modern plant has been developed to remove both these elements from liquor, but for economic reasons it is most practical to remove the bulk of the monohydric phenol together with a proportion only of the higher tar acids. It will be seen, therefore, that the production of concentrated ammonia liquor and the dephenolation of liquor make a substantial contribution to the effluent disposal problem and fall into place as intermediate steps in any chemically based system of effluent treatment. (See Fig. 1.)

The requirements of the plant for the production of concentrated ammonia may be summarised as follows:

1. The free ammonia content of the effluent should be no lower than that economically justifiable and necessary to meet disposal requirements. Typically, an effluent ammonia content of .03% (free) meets these requirements, and for crude liquors containing 1 to 2% free ammonia it represents a 3 to 1.5% ammonia slip. While reducing the slip is not difficult it becomes more and more uneconomic, since it requires a rapidly increasing steam consumption

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and/or capital expenditure. For a plant utilising a fixed number of still plates, this is shown graphically in Fig. 2.

2. The concentrated ammonia should contain 22 to 25% ammonia. This range represents the best compromise between diminution of product bulk to reduce transportation charges and the difficulties and losses associated with its loading, unloading and storage. Without taking special precautions, the ammonia loss on loading very concentrated liquor into a tanker, and consequent hazard to the attendant, can be surprisingly large. For example, an ammonia solution of 28% strength in the storage tank becomes 26% strength loaded in the tanker—an ammonia loss to atmosphere from 1,500 gal. of solution of 300 lb. in a period of 20 minutes.

3. The concentrated ammonia should be stable and free from solid carbonates and carbamates under all temperature conditions likely to be encountered. The formation of salt for a considerable period placed a severe limitation on the production of concentrated ammonia liquor, but modern decarbonating techniques have overcome this difficulty by simply removing the requisite proportion of carbon dioxide (and H_2S) before stripping the ammonia from the liquor. The decarbonating requirements may be judged from the $CO_2 : NH_3$ ratio of the liquor and the plant should be adequately equipped for this purpose.

4. The concentrated ammonia liquor should be despatched oil free. The quantity of light oil steam-distilled from the crude liquor shows wide variation, but invariably separates within the concentrated liquor storage tank. Means to withdraw the accumulated oil without interfering with the operation of the plant is therefore desirable.

5. The plant should possess the maximum availability and be under adequate automatic control. These requirements are inter-related in the sense that non-essential control equipment adds an avoidable maintenance burden and complication to the plant. For maximum availability, however, plants must be equipped with automatic control of the decarbonation and product strength, and the liquor feed system should be such that a reasonable constancy in rate of supply is attained.

The materials of construction should be such that excessive off-stream time for maintenance purposes is not necessary. In general a judicious distribution of cast iron and mild steel is all that is required, but a word of caution is necessary where liquors of high cyanide content are processed. The application of alternative methods of construction are discussed later.

With modern plants employing external dephlegmation, the most severe corrosion occurs in the vapour/liquor heat exchange equipment and the vapour condensing system. A form of construction of these units permitting ease of component part replacement is therefore desirable in any modern plant.

6. The plant should be flexible in operation and capable of handling crude liquor of varying composition without difficulty. The tonnage ammonia capacity of the plant and performance of the decarbonator are most significant in this connection.

High recovery efficiency

7. The plant should possess a high ammonia recovery efficiency so that the minimum quantity of distilled ammonia is recovered other than in the concentrated ammonia liquor product. All the ammonia recovered other than in the product suffers further distillation, thus increasing the overall steam requirements. For this reason, the use of direct water as a scrubbing medium is to be deprecated, since this directly increases the volumetric input to the plant.

8. The plant should be fitted with service meters so that comparative performance data may be obtained from time to time and an overall check of performance of the plant may be made without special effort.

9. Exhaust steam should be made available (from exhausters etc.) at a pressure greater than 5 p.s.i. The economics of concentrated ammonia liquor cannot bear the present day costs of high pressure steam.

The flow sheet of a typical plant with minimum instrumentation is shown in Fig 3 and an actual plant in Fig. 4.

The liquor should be delivered to the plant nominally

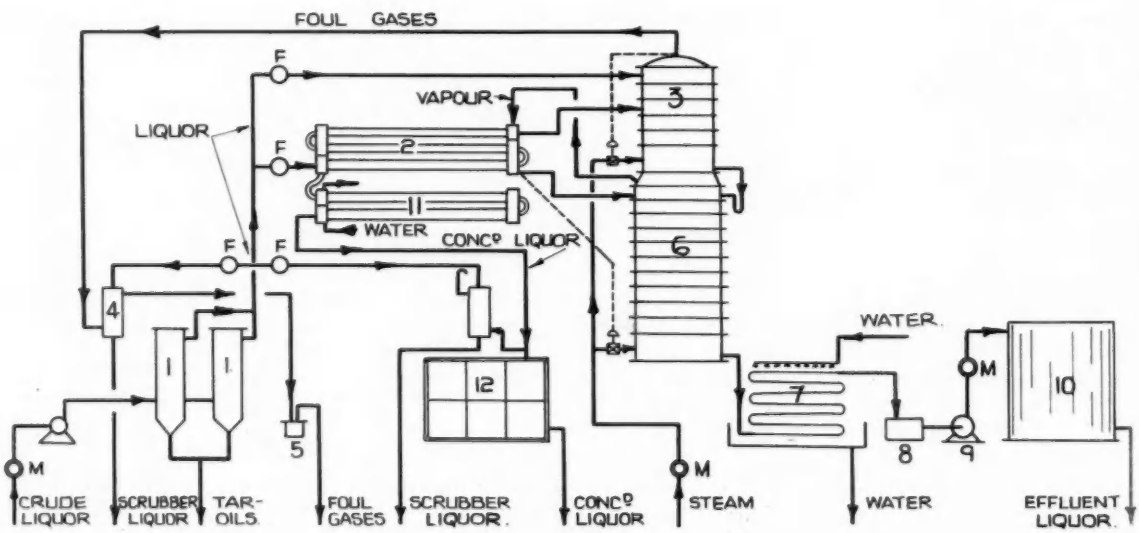

FIG. 3.

tar free, but to reduce light oil contamination duplicate filters (1) are provided. The bulk of the clean crude liquor to be distilled is preheated (2) before entry into the decarbonator (3) while the balance of the cold liquor is fed directly to the decarbonator head.

Decarbonation is effected by passing steam into the base of the decarbonator, the steam flow being automatically controlled to maintain a uniform decarbonator head temperature. The vapours leaving the recarbonator head are washed with crude liquor to recover ammonia in the foul gas scrubber (4), the exit gases preferably passing to the works foul main through a water seal (5) designed to prevent entry of air into the foul main under all circumstances.

The hot decarbonated liquor flows by gravity into the still (6) head and in its passage down the still is stripped with steam entering at the base to remove the ammonia and remaining hydrogen sulphide.

Cooling effluent liquor

The effluent liquor from the base of the still is normally cooled to approximately 100°F. in a cooler (7) of the rack or plate type arranged to operate under low liquor differential pressure so that pumping of hot effluent liquor may be avoided. The cooled liquor enters a small pumping tank (8) and is delivered by centrifugal pump (9) to the main effluent storage (10) prior to disposal.

Returning now to the still, the vapours emerging from the still head are partly condensed in a combination dephlegmator/crude liquor preheater (2). The dephlegmator section exists between the vapour inlet and the 'reflux' temperature control point, this latter temperature being maintained steady by automatic regulation of the steam flow to the still. Adjacent to the temperature control point a connection is provided to withdraw the condensate, conventionally termed reflux, which is returned to the still head. This mechanism represents a preferential removal of water from the condensing vapour stream thus substantially increasing the ammonia:water vapour ratio in the subsequent condensing system.

Final condensation and absorption of ammonia occurs in the water cooled condenser (11) from which the concentrated ammonia liquor flows by gravity into storage (12).

Some points of general interest are:

1. Electrically driven centrifugal pumps operating under moderately constant input head conditions deliver the liquor to the plant at a sufficiently uniform rate of flow to eliminate the need for automatic flow control.

Duplicate liquor filters

2. The liquor filters are duplicated so that the absorbed oil may be cleaned out at alternate intervals without interrupting the liquor flow.

3. Simple direct-in-line integrating meters (M) simplify overall plant performance checks.

4. Simple direct-in-line flow indicators (F) monitor important liquor flows.

5. The preheater-condenser is of concentric tube design to facilitate construction in cast iron and ease of examination and maintenance.

6. The still and decarbonator are of the bubble plate tube type constructed in cast iron.

7. The control temperatures are recorded; the controller recorders should be of high reliability to enhance plant flexibility. These instruments and their ancillaries must be conscientiously maintained and provided with adequate weather protection.

8. A second pen on each control recorder (to minimise chart changing) can advantageously record preheated liquor temperature and concentrated ammonia liquor temperature.

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9. The operating conditions of the crude and effluent liquor pumps are frequently sufficiently close to permit a single standby to be installed.

10. The foul gas scrubber (which should be carefully designed) should be operated with the minimum liquor flow compatible with ammonia recovery, so that re-absorption of carbon dioxide is restricted as far as possible.

11. The concentrated liquor should leave the condenser cold in order to minimise the load on the vapour scrubber and improve the ammonia recovery efficiency of the plant.

With a modern plant installed to make a 25% ammonia product and a hot effluent liquor containing .03% free ammonia, and operating on a typical crude liquor derived from conventional carbonising plant, the steam consumption is of the order of 20% by weight and the water consumption 25% by volume, of the crude liquor distilled.

Cooling of the effluent liquor demands additional water consumption, typically 200% of the volume of the crude liquor distilled when coolers of the plate type are employed. Rack coolers as frequently utilised exhibit wide seasonal variation in cooling water demand—nil to 100% of the crude liquor distilled.

Among special applications of this type of plant are (a) the production of semi-pure ammonia and (b) the desulphurisation of ammonia.

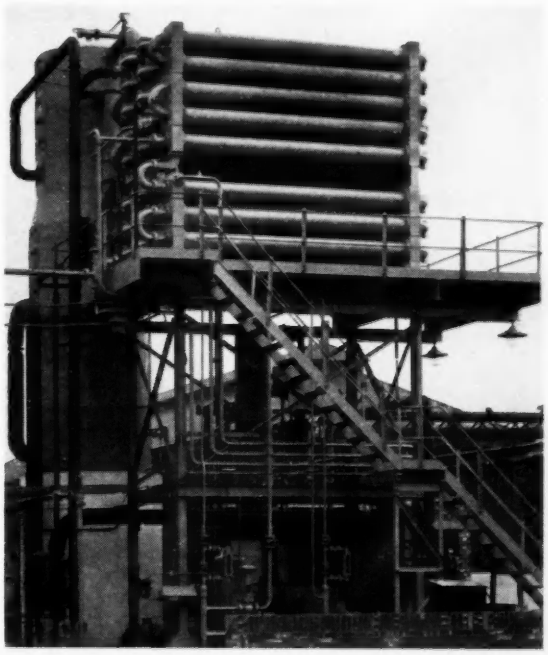

FIG. 4.

By greatly increasing the number of plates in the decarbonator (and as a further aid operating under pressure) decarbonation and desulphurisation may be increased to such an extent that an ammoniacal product containing less than 1% of CO_2 + H_2S may be obtained. For the corresponding purity at high ammonia strength—such as 30%—to be obtained in practice, however, the conventional con-

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denser must be followed by an efficient ammonia absorption system. This is due to the lack of CO_2 in the product which increases the vapour pressure of the ammonia in solution and makes its absorption much more difficult. Multi-stage counter current absorbers with stage cooling to abstract the heat of solution of the ammonia, have proved most efficient in practice and have enabled the ammonia to be recovered at high overall yield.

When reactions between ammonia and H_2S are employed to reduce the hydrogen sulphide content of gas during purification, the H_2S may be recovered in concentrated form and the ammonia reconstituted with plants of the above general type.

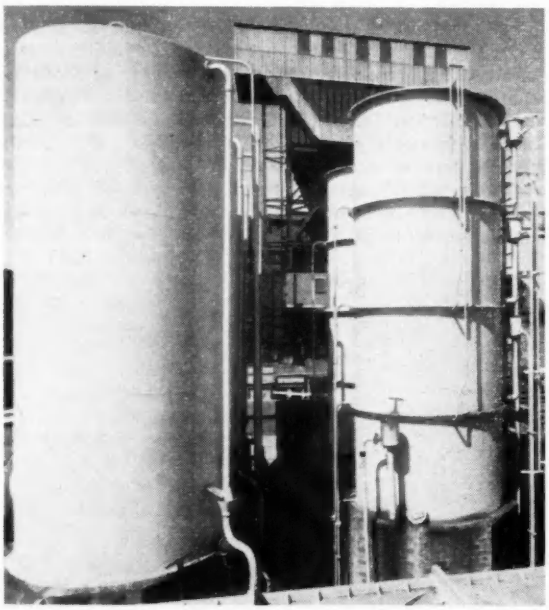

FIG. 5.

Corrosion is, fortunately, not sufficiently severe to prevent plant construction in cast iron and mild steel from being the economic choice in most cases as previously indicated.

When corrosion does occur it is thought to be associated with a high cyanide content in the crude liquor, which, on solution within the dephlegmating-condensing system, tends to destroy the protective sulphide film normally present on cast iron.

With modern plants employing external dephlegmation, therefore, corrosion tends to be concentrated in the dephlegmating-condensing system, parts of which may have an exceedingly short life. The still on the other hand is largely unaffected.

Aluminium alloys have been extensively investigated and those of high magnesium content (3 to 4%) have been found suitable for dephlegmator-condenser construction in many cases. However, when using aluminium or its alloys, great care must be taken to avoid direct contact with ferrous material because of the danger of electrolytic corrosion, which can be particularly severe at elevated temperatures.

Impervious graphite, glass and certain stainless steels have also been used to a certain extent and Epoxy resin coatings have shown very good resistance to concentrated ammonia liquor.

Many chemical and physical processes for the removal of phenolic constituents from liquor have been investigated, but most plants in current use are based upon liquid/liquid extraction principles. Only this form of plant will therefore be considered.

The choice of solvent is of primary significance, requiring good affinity for phenols yet permitting easy recovery of the phenols absorbed. It must be insoluble in the liquor and there must be an adequate difference in its density from that of the liquor. In general, the combination of these properties which will ensure a long life without deterioration, and low solvent losses.

It is hardly surprising that the ideal solvent has not been discovered, but creosote fractions have given very satisfactory service for the extraction of primarily monohydric phenols and more exotic materials such as butyl acetate, etc., for the extraction of primarily higher tar acids.

Monohydric phenol is the easiest to remove and a cheap solvent, such as creosote, is economically advantageous as long as the removal of the absorbed phenols can be carried out on the large scale. This has been demonstrated both at the Beckton and the Swan Village works where the phenolated oil is washed with caustic soda to concentrate the phenols as sodium phenolate; the latter is then treated along with large volumes of other phenolate in separate tar acid plants to recover the phenols and reconstitute the caustic soda.

Employing waste gases

At gasworks without integral or adjacent chemical works facilities, waste gases may be employed for springing the phenolate thus reducing the extent of the plant required to achieve dephenolation of the liquor with creosote type oil together with the production of crude phenols. The economic justification is very much a function of plant scale and effluent O.A./disposal charges, and thus demands an individual appraisal in each case.

Using creosote oil fractions, removal of more than 95% of the monohydric phenols and 40/60% of the higher tar acids may be readily achieved.

Specific solvents such as butyl acetate, phenosolvan, etc., exhibit better affinity than the creosote oil fractions in use to date for higher tar acids, and advantage of this fact has been taken to treat the relatively small volume of retort house liquor for the removal of higher tar acids, thus markedly reducing the O.A. value of the works effluent.

Recovery of the phenols from the solvent is by distillation in this case, as with most other volatile solvents. The plant is thus relatively simple and self-contained, but suffers economically from the high cost of the solvent which renders process losses particularly significant. Continuous losses result from the slight solubility of the solvent in the liquor and the operation of the distillation stage. Since this is fundamental, it cannot be avoided.

Quite the contrary is true of the creosote oil processes where oil losses are comparatively insignificant, but greater attention to the design of the individual extraction stages is necessary in order to achieve a high overall extraction efficiency. A good example of this aspect is the installation at the Swan Village works where three continuous countercurrent pump mixing stages are employed to contact the liquor and oil in a very intensive manner. Part of this plant is shown in Fig. 5.

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One of the main disadvantages in the burning of spent oxide to produce acid is that the iron oxide residue produced is fairly light and finely divided. Considerable quantities are carried forward with the gases and can cause blockages in the packed towers in the plant and also contaminate the acid produced. In an attempt to reduce this dust problem the gas passes through a large brick flue system where the majority of the dust can settle out. Cyclone dust extractors and electrostatic precipitators are also used on some plants where gas conditions are suitable.

Since this paper mainly deals with the use of spent oxide in acid manufacture, only brief mention will be made of the reactions that take place with the gases produced from the burner. Because the gases are contaminated, and extensive purification would be costly, almost all spent oxide is used in chamber plants. These plants depend on the use of oxides of nitrogen from the catalytic oxidation of ammonia, to oxidise the sulphur dioxide to sulphur trioxide; the nitric oxide from this reaction being re-oxidised back to nitrogen dioxide by the oxygen present in the gases.

Re-oxidation of nitric oxide

This takes place mainly in the lead chambers which are large enough to allow sufficient time for the reaction to proceed to completion. Water sprays are situated in the roofs of the chambers to absorb the sulphur trioxide and form a weak 60% chamber acid. This weak acid flows down the Glover tower situated immediately after the burners and dust settlement flues, and the hot burner gases passing up the tower concentrate the acid to 77% which, after cooling, is the acid sold. The oxides of nitrogen are recovered at the end of the plant in Gay Lussac towers by absorption in cold 77% acid, and when this 'nitrous vitriol' is fed from the top of the Glover tower, the rise in temperature releases the nitrogen oxides which pass back into the gas stream for re-use.

The rate of production of sulphuric acid is controlled by the rate of the re-oxidation of the nitric oxide produced. This reaction is an unusual one in that it has a negative temperature coefficient of velocity which means that the rate of oxidation is increased by cooling the gases. The size of the lead chambers required has been reduced on some plants by replacing them with Mills Packard towers which are water cooled on the outside to obtain this increased reaction rate. Tower units are also in operation which use a much higher concentration of nitrogen oxides to reduce the size of the plants required.

The acid for sale produced from the Glover tower is known as brown oil of vitriol (B.O.V.) and contains 77% by weight of sulphuric acid. A typical analysis of this acid is:

Sulphuric acid	77% w/w as H_2SO_4
Iron (present as $Fe_2(SO_4)_3$) ..	.09% w/w as Fe_2O_3
Aluminium (present as $Al_2(SO_4)_3$)	0.5¾ w/w as Al_2O_3
Lead (present as $PbSO_4$) ..	.04% w/w as $PbSO_4$
Oxides of nitrogen	.01% w/w as HNO_3

The acid is saturated with ferric sulphate derived from the dust passing forward from the burners. Ferric sulphate is also present in suspension and will continue to be deposited from the acid on storage. This is allowed to settle out before the acid is despatched, which necessitates the cleaning out of the stores from time to time. The greatest variable in the above analysis will be the aluminium sulphate since it is fairly soluble and does not saturate the acid. The amount present varies with the source of the spent oxide and the efficiency of dust settlement. The acid produced is mainly used for fertiliser manufacture, both as phosphates and ammonium sulphates, and for metal pickling.

The burnt residue from the burners is carried away by a screw conveyor to a storage hopper from which it can be loaded into lorries. It consists mainly of iron oxide but will contain silica and calcium sulphate. If the oxide has been subjected to high temperatures in the burning operation, then it is rendered fairly inactive for re-use as a purifying medium at the gasworks. Some is remixed with wood, etc., and re-used, especially the material obtained from the Flixborough burner which operates at a slightly lower temperature. Further outlets for this material are the blast furnaces, and some is used in the cement trade.

The production of spent oxide in this country is falling slowly, probably due to the increased production of water gas which has a lower sulphur content. Also with the future use of total gasification methods for the production of gas, the sulphur may be available as a by-product in a different form, possibly some being recovered as elemental sulphur. Any change-over that may take place in producing the by-product sulphur compounds in different forms from spent oxide will naturally be a gradual process, and consequently it is likely that the chamber process will be in use for many years yet, producing commercial sulphuric acid from spent oxide.

For "SLIP" H_2S control
use ZINC ACETATE
Supplied by
FREDERICK ALLEN & SONS (POPLAR) LTD.
PHOENIX CHEMICAL WORKS, UPPER NORTH STREET,
LONDON E.14 Tel. No. EASt 2673
FULL LIST OF PRODUCTS ON APPLICATION

J. J. PRIESTLEY : continued from p. 395

sulphur entails two further stages, namely a pressure autoclave treatment of the filter cake in which the temperature is high enough to melt the sulphur and separate it from the water, and secondly a distillation stage. The molten sulphur from the autoclave is pumped into a retort in which the sulphur vapours are distilled off and the organic hydrocarbons destroyed. The sulphur leaves the condenser as a clear yellow liquid and runs to a pig-casting machine where it cools to solid yellow blocks.

Those processes using aqueous suspensions as purifying media, such as the States-Mines and Manchester, not only suffer fouling of the sulphur by tarry bodies, but also by the solid reagent, some of which is always so closely associated with the sulphur as to be frothed off with it. The purification of such crude sulphur is exceedingly difficult and has never been carried out successfully on a large scale. The Manchester process has the great advantage of being able to purify gas to below referees standard, requiring no auxiliary fine purification as most other liquid processes do, yet the intractable nature of the sulphur produced, particularly its iron oxide and tar contents, has proved to be one of the most serious disadvantages against the wider adoption of the process.

In the second group of liquid purification processes, which could be termed the absorption-desorption group, the hydrogen sulphide is not oxidised to sulphur, but is absorbed in a suitable reagent, and afterwards driven out of solution by heat. Reagents in present use for example, are certain ethanolamines, ammonia and potassium carbonate. The process is not one of simple physical solution since hydro-sulphides and sulphides are formed, dissociation by heat, with or without the use of vacuum, yielding the original hydrogen sulphide.

Control of absorption

The reagents are basic, hence other acid gases, notably carbon dioxide and hydrogen cyanide, are also absorbed. By careful choice of washing conditions and limiting the time of contact of the reagent and the gas, the carbon dioxide pick-up can be reduced to a minimum and the concentration of hydrogen sulphide increased to a maximum. It is from this point that the production of pure sulphur begins.

The crude hydrogen sulphide flow is split into two streams, one being a third, the other two-thirds, of the total volume. The smaller stream is burned to form sulphur dioxide, the heat of combustion being utilised for steam raising by carrying out the operation in a boiler designed for the purpose. The flue gas, rich in sulphur dioxide, is then mixed with the original two-thirds of crude hydrogen sulphide at the inlet of a Claus kiln. This consists of a chamber containing two alumina catalyst masses in series and operates at a temperature of about 300°C. Elemental sulphur is formed according to the reaction:

$$2H_2S + SO_2 \rightarrow 2H_2O + 3S$$

and drains away from the kiln as a liquid which is cast into blocks. If the crude hydrogen sulphide used in the process contains ammonia, a high temperature pre-treatment is used before the Claus kiln reaction, the object of which is to crack the ammonia, otherwise it would form ammonium sulphate in the catalyst masses. The recovery of pure sulphur in this type of process is about 80% of that present in the original gases. The quality of the recovered sulphur is very high, and is usually of the order of 99.95% S.

None of the processes in this group is cheap, one of the major operating costs being that of steam for stripping out the acid gases. Cyanide corrosion in the stripping stage can also be very troublesome, as was experienced for instance on the Collin plant at Corby using ammonia.

From the gas engineer's point of view the absorption-desorption processes are at a disadvantage in that they are all incapable of cleaning the gas of hydrogen sulphide down to the referees' standard.

Oxide catch boxes must be installed downstream from the liquid purification plant for removing the remainder of the unabsorbed hydrogen sulphide, which may be quite high. i.e., 200 grains per 100 cu.ft., with some types of plant, but with others only a fraction of this amount.

The production of sulphur dioxide instead of sulphur is hardly to be considered a worthwhile alternative. If the production of the pure gas is set aside as impractical, the alternative is sulphuric acid. Plants for this duty in gas works would be very small, for instance a works producing 20 million cu.ft. gas per day, would require a sulphuric acid plant having a production capacity of only 30 tons per day.

There is no technical difficulty in producing pure sulphur from carbonisation and similar gases. The main reason why this is not practised more widely is one of cost when compared with the low world price of sulphur, which again is due to the plentiful supply from the Gulf Coast of the United States.

Colour scheme for pipes

A RELIABLE colour-identification scheme for pipelines is essential in countless spheres of industry. Neither safety nor efficiency is assured where labyrinths of pipes abound, their contents often enigmatic until someone —perhaps an unsuspecting maintenance engineer—becomes the victim of an accident.

Hence the importance of the revised British Standard for Identification of Pipelines (B.S. 1710: 1960) with its recommendations for an identification colour code. It applies to pipelines in buildings, industrial installations and on water and land transport.

British Standard ground colours are specified to distinguish between the various classes of contents—white for air, canary yellow for town gas, dark grey for chemicals and so on. Colour bands are specified to individualise within classes; for example, a pipe containing butane would be dark grey, with a red band.

The 16-page standard contains five pages of fully annotated colour diagrams. Copies of this standard may be obtained from the British Standards Institution, Sales Branch, 2, Park Street, London, W.1, price 6s. (Postage will be charged extra to non-subscribers).

Guide to fan engineering

A SECOND and enlarged edition of 'Wood's Practical Guide to Fan Engineering' has recently been published. Since the first edition was published in 1952, it has been translated and published in French with the aid of Maison Dunod of Paris.

The demand for fans continues to expand as more and more energy is contained in less and less space. This is particularly true of electronic engineering in which fans are being increasingly used, as they are in cooling aircraft and rockets. The new edition has been brought up to date and sections on drying, air heating, noise and vibration have been included, the section on noise control being completely rewritten.

It is published by Woods of Colchester Ltd., price 17s. 6d

GAS ON THE CONTINENT

Wide range of papers at French gas congress

THE 77th Congress of the Association Technique du Gaz en France was held in Paris from June 27-July 2.

A recent issue of the *Journal des Industries du Gaz* contains a list of the 36 technical papers which were presented. The issue also contains Chapter 10 from M. Bolzinger's new book on chemical engineering and the treatment of gas which is to be published shortly. It deals with the absorption of naphthalene and benzole by tar in the course of condensation and the protection of pipe connections and works apparatus against deposits of naphthalene and tarry 'mud.'

The treatment covers oil-gas as well as coal-gas. 'When one is producing,' the author says, 'a rich oil-gas (950 to 1,000 B.t.u. per cu.ft.) there is much tar and little naphthalene and the case is similar to that of continuous verticals: When oil-gas at 470 B.t.u. per cu.ft. is produced by the cracking of heavy oils there is, on the contrary, little tar but much naphthalene, and the case is similar to that of coal-gas production in coke-ovens.'

A tunnel kiln at Digoin 'with naked flame,' using natural gas for the burning of glazed stoneware, is described by M. P. Pirion, Manager of the pottery company there. The article is well illustrated by drawings and photographs.

A number of devices for the control and security of domestic gas appliances, cookers, water heaters, space heaters, etc., are described by M. Roger Brisou attached to the regional staff of the joint electricity and gas industries at Lyons. Some of these devices are electrical.

An event claimed to be of importance in the history of the industrial development of Algeria is the signing of a convention by the companies exploiting the natural gas deposits at Hassi R'Mel and Electricité et Gaz d'Algérie defining the transport of gas from the wells to the coast and the commercial use of the gas.

A line for a 24-in. pipe is being planned from the oil- and gas-producing region to a point near Mortaganem (the port which is suggested as the departure point of the ultimate submarine pipeline to Spain) and from there west to Oran and east to Algiers. It is hoped that this project will provide cheap energy for many new industries in Algeria.

Gas output by Gaz de France for the month of March showed an increase of 11.7% over that of the same month last year. Raw materials used showed a decrease of 10.8% in coal with an increase of 15.7% in liquid petroleum products.

Plant for reforming propane with steam

THE realisation on an industrial scale of a plant for reforming propane with steam by the P9 process announced by the research department in April 1959 has met with success.

The thermal efficiency is about 88% for a final gas of 4,200 calories (440 B.t.u.) completely interchangeable with town gas and obtained by dilution of gas completely cracked, or by utilising the air of reaction so as to obtain a hydrogen content of about 45% after enrichment with propane. Apart from the low cost of installation, the plant at Le Mans shows how rapidly it can be brought into action, gas of normal characteristics being obtained in 1½ hours from cold and full nominal capacity attainable in two hours.

The Le Mans plant is being adapted to work on light oils.

Work on the establishment of significant data on the physical, chemical and mechanical properties, and the catalytic activity of the catalysers, both new and used, sent to the research department for examination, is now complete.

Quite recently a small set of apparatus based on the American Catatest was set up in the laboratory for the purpose of studying, by a continuous, externally heated process, the reforming of gaseous hydrocarbons on small quantities of catalysts (about 50 cu.m., a quantity only 1/100 of that required by the experimental apparatus previously in use).

This apparatus, of which the first tests appear to be promising, will permit an evaluation, under closely defined conditions and on small samples, of the activity of the different catalysts used by Gaz de France. The rapidity of operation opens up an interesting field for investigation and promises the possibility of a sizeable reduction in the time required to deal with certain problems which are put to the research department.

Space heating by radiant heaters

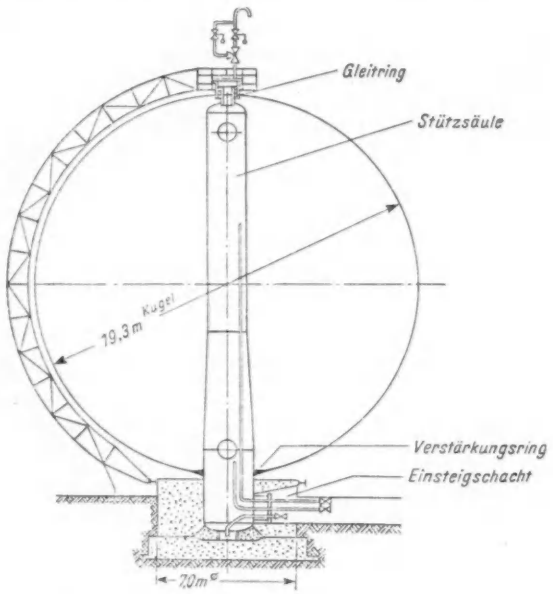
GAS-FIRED radiant heaters emit their heat energy in wavelengths within a range of from .8 to 10 microns and can therefore be classified as infra-red heaters. If the temperature of the radiant heater is kept at between 800 and 900°C., its radiating capacity is 155 to 230 B.t.u. per sq.in. per hour of radiating surface area, so that a gas-fired heater of 46 sq.in. radiating area gives off between 7,130 and 10,580 B.t.u. per hour.

For the purposes of heating workshops and large halls, such a size of heater is considered adequate for a floor area of 85 to 105 sq.ft. and in exceptional cases even up to 200 sq.ft. For heights of less than 13 ft., radiant heaters are as a rule only recommended for open halls or where individual working places have to be heated. The usual vertical radiant heat sources are recommended for heights above 13 ft. with a nominal spacing of twice their height from floor level.

The gas rate can be taken as 28 to 42 cu.ft. per hour per heater at a gas pressure of 2½ to 4 in. w.g. and a calorific value of 427 B.t.u. per cu.ft.—*J. Walter. Gaswärme*, 1960. **9**, 123.

New type spherical gasholder

WHEN a new gasholder was required for works extension at the Badische Anilin Co. in West Germany, which receives its gas from the Saar district, a new type of spherical gasholder was chosen. It has a diameter of 63 ft. 4 in. and a volume of 132,000 cu.ft., which corresponds at a working pressure of 114 p.s.i. to a free gas volume of 1.056 mill. cu.ft.



Key
Gleitring . . . Sliding ring.
Stützsäule . . . support column.
Verstärkungsring . . . reinforced support ring.
Einsteigschacht . . . manhole.
7.0 m ϕ . . . 23 ft. diameter.
19.3 m Kugel . . . 63 ft. 4 in. dia. sphere.

The spherical shell is built around a central support column and the bearing pressure is transmitted through a reinforced support ring. The shell can expand relative to the column along a sliding ring at the column top. The material chosen for the shell is a fine-grained steel to the German specification BH38K of $\frac{10}{16}$-in. thickness.

The 88 individual sectional plates were welded on site and each weld was inspected by a gamma-ray test. The erection took four months. Strain gauge measurements on the complete gasholder under pressure have confirmed that the actual stresses lie below the calculated permissible stresses. This gasholder has now been in service for nearly two years and has given full satisfaction.—*O. Schulze-Dirks. Das Gas-und Wasserfach.* 1960, **101,** 267.

Pumped circulation central heating

CENTRAL heating systems of this type have been appreciated for some years in Switzerland, on account of their low initial cost, their compactness and speed of control. A typical unit comprises a main burner, protected by a magnetic valve and a bimetal controlled valve. An igniting flame lit by the pilot flame, acting on the bimetal, ensures that gas is not supplied by the main valve when the pilot flame is not burning. A thermostat at the outlet from the water coil acting on the magnetic valve protects the former against overheating. The flow of gas to the main burner is controlled by a valve working on the rotameter principle. If the control device indicates that heat is required by the central heating system the pump is switched on and the magnetic valve is opened. Gas flows to the igniting flame and operates the bimetal to open the main gas flow in approximately 30 seconds.

Automatic control can be arranged by a variety of means. First, the water temperature at the outlet from the heater or at some convenient point can be used for control, the pump running continuously. This is the cheapest solution and has certain clear advantages. Alternatively, control can be by a room thermostat, the heater being arranged to provide water at a temperature set by the gas and water flow rates. This provides a quicker heating-up rate and can be controlled by a clock to suit day and night requirements. The most elegant solution is to use an outside thermostat to control the water temperature.

The rate of heating is somewhat affected by the outside temperature, but the clock can be adjusted to counteract this. To avoid overheating, the pump is always arranged to run for at least a short period after the main flame is shut off. A. Brennwald, *Schweizer Verein von Gas- und Wasserfachmännern,* 1959, **39,** 219.

New type of warning lamp

MESSRS. Adolf Nissen of Tönning (Eider) in West Germany have put on the market a new type of warning lamp for road blocks guarding excavations for the laying of gas pipes, etc. The novel feature of this lamp is that it combines a continuous amber light with an intermittent flash of extremely high brilliancy which is visible for several miles. The beams of light are directed both towards the oncoming traffic and in the opposite direction. Single lamps are not adequate to warn traffic on fast motor roads and the same firm supplies sets of three to five lamps mounted in a row on a tubular barrier with feet on either end.—*Das Gas-und Wasserfach,* 1960, **101,** 411.

Determination of tar in crude sulphur

VARIOUS methods are in use for finding the tar content of crude sulphur obtained in the extraction of spent purifier oxide with carbon bisulphide. None of them is completely satisfactory, especially when the tar content exceeds .2%.

In the new method, the adsorption affinity of the tarry substance is increased by a preliminary treatment of the crude sulphur with concentrated sulphuric acid during which the tarry substance is polymerised or sulphonated. The pure sulphur content is then determined and the tar contained in the sample is obtained as the difference between crude sulphur and the sum of pure sulphur and ash.

Silica gel is used as the adsorption medium instead of activated carbon because the appearance of black colouring makes the adsorption process visible and controllable. One determination takes about 90 minutes and control analyses show good agreement to within .03% at a nominal 2.025% of tarry substance.—*S. Schubert. Das Gas-und Wasserfach.* 1960, **101,** 367.

COOKING SAFER BY GAS—REPORT

ELECTRIC cookers are three times more likely to cause fires in domestic premises than gas cookers, a report by the Department of Scientific and Industrial Research and the Fire Research Board shows.

A comprehensive analysis shows that in 1956 there were three to four gas cookers in use for every electric one, but only 17% more fires originated in gas cookers.

It is held that there is a greater likelihood of food igniting in electric cookers, while gas caused a greater proportion of the fires associated with clothing.

Fires in the United Kingdom in 1960 notified to the Fire Research Station to the end of August were 85,761. Heating apparatus caused outbreaks as follows: Coal 4,943; Electricity 553; oil 1,477, and gas 44.

Let's turn on the central heat . . .

A GAS COUNCIL central heating campaign will be launched by mid-September. The budget is substantial and covers national daily and Sunday newspapers, and periodicals. The campaign will be backed with advertising by Area Gas Boards.

Coverage is as follows: Daily Press—Daily Express, Daily Mail, News Chronicle, Daily Telegraph, The Times, The Guardian, The Scotsman. Sundays—Sunday Express, Sunday Dispatch, The Observer, Sunday Times. Periodicals—Radio Times, Punch, The Listener, The Queen, Homes and Gardens, House and Garden, House Beautiful, Ideal Home, Reader's Digest, Good Housekeeping, Woman's Journal.

The campaign will last from mid-September to the end of March. In newspapers it will take the form of full-column triples, four-colour pages in magazines, and black/white pages in Radio Times and The Listener.

The campaign will emphasise the recently introduced 'Personal Loan' scheme for purchasing and installing gas and coke central heating systems and the advantages of using gas as a fuel for central heating.

On right lines

If gas can remain competitive there will continue to be a great many domestic consumers and industrialists who prefer it for their various needs, Mr. Richard Wood, Minister of Power, said during an informal visit to Litchurch Gas Works and Cavendish Street Depot, Derby, in Notts. and Derby Division of the East Midlands Gas Board.

Mr. Wood said: 'I have been particularly impressed with what has been done at the development centre to fit in with the needs of industrialists, and with the very modern appliances of the housing centre. The kind of thing the East Midlands Gas Board is doing seems to be exactly on the right lines.'

. . . and the councillors see for themselves

The Wales Gas Board has invited six members of Caerleon (Mon.) Council to travel to Harlow New Town to study the central heating system used in the houses there.

The outcome of the visit will decide the type of central heating which the council will use in five-bedroomed houses on their Lodge Farm Estate.

Seaguard Ltd., the cathodic protection specialists, have moved to: Seaguard House, 20, Balcombe Street, London, N.W.1. Telephone: AMBassador 1614/8.

COMING EVENTS

September 15-16. — SCOTTISH ASSOCIATION OF GAS MANAGERS: Largs. Annual General Meeting.

September 21.—SOUTH WESTERN G.C.C.: 33, Bridge Street, Taunton. 11.0 a.m.

September 27.—THE COMBUSTION ENGINEERING ASSOCIATION, NORTH WEST REGION: The Engineers' Club, Albert Square, Manchester. 'Modern Fuels and Their Application,' by D. C. Gunn. 2.30 p.m.

September 27.—THE SOCIETY OF INSTRUMENT TECHNOLOGY: Manson House, 26, Portland Place, London, W.1. 'Gas Sampling for Automatic Combustion Control,' by R. Hardy and 'Smoke Density Measurement,' by G. H. Laycock. 7.0 p.m.

NEWS SUPPLEMENT

STEELMEN TO HAVE NEW COKE PLANTS

TWO new coking plants together valued at over £4m. are to be built by Woodall-Duckham Construction Company Ltd.

Colvilles Ltd. have ordered two more batteries of coke ovens with complete coal and coke handling systems and a by-products recovery plant for their Ravenscraig works.

This new plant will have a coke output capacity equal to that of the plant at present under construction so that, by the end of 1962, when all six batteries will be in operation, the Ravenscraig works will be processing 1¾ million tons of coal annually.

Demolition

The other new plant is for the United Coke and Chemicals Co. Ltd. (a subsidiary of the United Steel Companies Ltd.) at Orgreave.

The contract includes the demolition of a derelict battery of ovens and the building of a new battery of 43 ovens with by-products recovery and benzole plants and a gas boosting plant.

Demolition work is already in progress and the building of the new installation will be completed by the end of 1962.

Blast furnace contract

ASHMORE, Benson, Pease & Co. Ltd. (of the Davy-Ashmore Group) has received instructions from Colvilles Ltd., to proceed with the design, manufacture and erection of a third blast furnace plant for their Ravenscraig Works. Value of the contract is in excess of one million pounds.

While the plant will be similar to the No. 1 blast furnace which Ashmore, Benson, Pease & Co., Ltd., completed for Colvilles in 1957, and also to the No. 2 furnace which the same company is at present constructing at Ravenscraig, it is of slightly increased capacity, and will incorporate changes in the design of brick lining and water cooling, in accordance with recent trends in blast furnace practice.

UPWARD TREND IN GAS SALES

IN their bulletin to the Southern Gas Consultative Council for the quarter ended June 30, 1960, the Southern Gas Board reported an improvement in gas sales which it was hoped would be maintained. Increases in gas sold to industrial and commercial customers showed a steady upward trend.

The re-imposition of Government restrictions on hire purchase on April 29 had an immediate impact on appliance sales. Hire purchase sales were almost 25% less than during the same quarter of 1959.

Even so, sales of fires, radiators and refrigerators increased. Results are considered favourable in a period of credit restrictions.

From refinery

By June, refinery gas supplied to Southampton Works from Fawley Refinery rose to a level of 82,000 therms per day. Town gas now produced from this in either carburetted water gas plants or in catalytic oil gas plants amounts to 22% of the Board's total production.

The foundations for the concentrated ammoniacal liquor plant at Portsmouth were completed during the quarter, and work started on a similar construction at Southampton. Both plants will assist in the production of ammonia and nitrogenous liquor for agricultural purposes, the demand for which has grown from 202,000 gal. in 1950/51 to nearly 10,000,000 gal. in 1959-60.

Transfers

Transfers of gas from Southampton were increased, approximately 5.8 mill. cu.ft. a day being moved to Portsmouth and 5.3 mill. cu.ft. to the Reading Region. About 4 mill. cu.ft. of the gas sent to Reading has been passed through Basingstoke to the Aldershot district.

Connections have been made from the backbone main to the local distribution systems at Burley, nr. Ringwood, and at Thatcham, Berks, giving much improved gas pressure in the two districts.

The new gas drying plants have much improved the condition of services. The net increase in services laid was 3,337.

Old lanterns price move

THE sale of old gas lanterns in Leeds has been suspended by the city's Street Lighting Department until after their next meeting, when the selling price is likely to be reviewed if the lanterns are to be sold as a commercial proposition.

The lanterns, which are copper under green paint, look attractive when polished. They have been selling briskly.

The suspension came after a Lincoln businessman, who read in a Leeds evening paper that these lanterns were selling as attractive ornaments in the home, made a bid for a large number.

Butane/air plans in the north

THE Northern Gas Board are in course of installing a butane-air plant to replace the horizontal retorts at Stanhope, Co. Durham.

Situated in the remote north-westerly corner of the Board's Darlington Division, this works is too far away for it to be economic to lay a pipe connecting it to an existing grid supply.

This is in contrast with the other small undertakings in the division at Bedale, Masham, and Leyburn, which are now supplied from the Board's grid main.

The plant at Stanhope will be a Holmes-Mallet capable of supplying 4,000 cu.ft. of butane air mixture per hour.

A similar plant was installed some time ago at Alston, in the Board's Cumberland Division.

PERSONAL NOTES

MR. R. W. F. BROWN, for the past three years manager for the East Midlands Gas Board at Melton Mowbray, Leics., is moving to Loughborough where he has been appointed the Board's North-West Leicestershire District Manager. He succeeds MR. J. A. DEMONT, who has been at Loughborough for the past two years and who is now going to Northampton.

MR. J. R. MOORE, manager of the Fisher Governor Company at the Rochester factory of the Elliott-Automation Group, has been appointed Assistant General Manager of the Elliott Valve group of companies.

COUNCILLOR W. A. TRAIN of Sharnbrook, Bedfordshire, has been appointed a member of the Eastern Gas Consultative Council, by the Minister of Power.

MR. W. B. CLARIDGE, Regional Manager at Swindon, will be retiring at the end of September after 50 years' service in the industry. He began his career in the gas industry with Harpenden in 1910, moving six years later to Farnham Gas and Electricity Company. After serving in the Royal Navy during the first world war, he moved to the managership of Marlborough Gas Company, where, in 1935, he became 'integrated' with Swindon Gas Company, and was appointed manager of Swindon Division, which post he held until his appointment as Regional Manager last year. On his retirement Mr. Claridge will hand over his duties to MR. T. L. TALLENTIRE, who will then be Regional Manager for Cheltenham, Gloucester, Bath and Swindon.

MR. C. H. LEACH has been re-appointed Chairman of the Southern Gas Board by the Minister of Power.

NEW ASSISTANT EDITOR

When MR. COLIN J. GODBOLD relinquished the post of Assistant Editor in order to take up an appointment in Canada, the 'GAS JOURNAL' invited MR. WALTER F. TRUMAN to return to this country from S. Rhodesia, where for the past two years he has been a senior sub-editor on the Evening Standard, in the Federal capital, Salisbury.

He commenced his duties with the 'JOURNAL' on September 12.

No stranger to gas journalism, Mr. Truman worked over four years on another journal in the field and has a considerable knowledge of the industry.

He was earlier for over six years a sub-editor on the Natal Daily News, an evening newspaper published in Durban, S. Africa, and while in the Union also spent several months as a sub-editor with the South African Press Association in Johannesburg.

Mr. Truman has also worked in Southend-on-Sea, Essex. He underwent his early training on the North-Western Evening Mail at Barrow-in-Furness.

Oblique valve

THE new oblique air-operated valve, the description of which appeared in the Technical Development Section on p. 240 of our issue of August 17, was erroneously stated as being manufactured by Winn & Coles Ltd. This should have read Charles Winn & Co. Ltd. We apologise to the firms concerned for this error and to our readers for any inconvenience they may have experienced.